XRS-FP QUICK START (FOR EXPERIENCED USERS)

Amptek's products provide a very powerful tool for quantitative XRF analysis. The most accurate analysis requires finely tuning many different parameters, particularly in the analysis software. It also requires careful spectroscopic calibration using spectra measured from samples with compositions close to that of the material under test.

Fortunately, for an approximate result, a "standardless" analysis may be used and many of the parameters are not critical. This Quick Start guide is intended to help the first time user of the XRS-FP software to get some initial results, as a starting point for tuning parameters. This guide assumes the user is familiar with Amptek's XR100 detectors, PX4 signal processor, Mini-X X-ray tube, ADMCA software and has used these previously. Each has its own User Manual and Quick Start Guide, supplied separately.

The key steps are (1) setting up and configuring the hardware (XR100/PX4 and Mini-X), (2) calibrating the energy scale, (3) launching and configuring the analysis software, (4) acquiring a spectrum from the object under test, and (5) running the analysis. The following guide lists the steps in a little more detail and includes links to even more detailed guides.

WARNINGS AND PRECAUTIONS

The Mini-X generates X-ray radiation during normal operation and presents a safety hazard. It must be used with radiation shielding (not supplied by Amptek) and operated by qualified personnel.

RADIATION SAFETY IS YOUR RESPONSBILITY!!

High voltages are present in the Mini-X, the XR100, and the PX4. The Mini-X voltage (up to 40 kV) presents a potential personnel safety hazard.
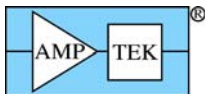
The detector and X-ray tube both contain thin, fragile Be windows. If either window is damaged, the unit will be destroyed, cannot be repaired, and is not covered under warranty. Do not touch the windows!

1. **Set up the acquisition system**

   1.1. Mount the XR100 detector, PX4 digital pulse processor, and the Mini-X onto your fixture. A 40 mil (1000 μm) Al filter on the Mini-X tube is often a good starting point. Make all appropriate electrical connections. See the Set-Up Guide section of this document for additional information on mechanical and electrical setup.

   1.2. Install the three software packages. ADMCA is used for data acquisition and control of the PX4, Mini-X is used to control the Mini-X X-ray tube, and XRS-FP is used for quantitative analysis.

   1.3. If you are new to these products, please refer to the Quick Start Guides which are included on the installation CD. These provide details on software installation and electrical setup.

2. **Configure the acquisition and control software**

   2.1. Launch the Mini-X software. We recommend starting with HV on 30 kVp and current of 15 μA. Make certain your radiation shielding is in place, then turn on the tube and use a radiation survey meter to verify that your shielding is adequate. You will probably want to increase the current later, to get a higher count rate for faster measurements, but start low.

   2.2. Turn on the PX4, launch the ADMCA software, and connect to the PX4.

   2.3. Configure the PX4.

   - If you do not know how to configure your detector, we recommend starting with one of the standard "Amptek Detector Configuration" options. It will enable one to take a spectrum quickly and provide a good starting point for tuning.

   - There are many parameters which can be tuned to get the best performance, but the most critical adjustment is the gain, which must be set to cover the full energy range to be measured. Use the ">" and "<" buttons as a quick gain adjustment. If running at high count

rates, we recommend using a short peaking time. After these parameters are set, remove the excitation source and "tune" the thresholds using the "auto-tune" button on the ADMCA toolbar.

2.4. Take a calibration spectrum and perform an energy calibration.

- We recommend using at least two different photopeaks of known energy and spread across the energy range of interest, with at least 50,000 counts in each photopeak.

- This guide assumes use of the "Calibrate" function within ADMCA. See Energy Calibration for additional information on ADMCA's calibration. The FP manual describe calibration in FP. The calibration needs to be in units of keV, not eV, for compatibility with FP.

2.5. Launch FP

2.5.1 Click the "FP" button on the ADMCA menu bar. A small box will appear, asking to "Start XRS-FP". Click "Yes". See Launching XRS-FP for screen plots.

2.5.2 A yellow box will appear with version information etc. Click anywhere in the box.

2.5.3 A gray box will appear. Click "Expert" mode and the "Main Screen" appears.

3. Configure the analysis software

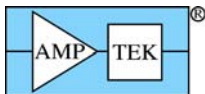3.1. Set-up the key parameters.

The XRS-FP "set up" menu contains several items that bring up their own dialogs, each with many parameters. Many of the options may not be relevant for a particular system. Only a few are vital for an approximate analysis but the most accurate analyses will result from careful settings.

3.1.1 MCA: Set the number of channels to match that selected in ADMCA for the PX4. Set the "Range" to the value nearest the energy of the maximum channel in the PX4. For example, if the calibrated PX4 yields 35 keV full scale, set "Range" to 40 keV.

3.1.2 Detector: The table below shows default parameters for the six most common Amptek detector configurations. The data sheet supplied with the unit will show the configuration. Note that the "area" in this table is the area of the collimator, for units with an internal collimator. Note also that the "resolution" in this table is a typical value under Amptek's standard test configuration. We recommend measuring it for your detector, setup, and configuration and using this measured value.

Description		Detector Type	Detector Z	Area	Thickness	Gold Layer	Dead layer	Window type	Window Thickness	Al window coating	C window coating	Resolution at Mn Ka
					um	um	um			um	um	eV
SDD	7 mm2/450 um	5 (SDD)	Si	4.4	450	0.00	0.15	Be		0.0	1.0	145
SiPIN	6 mm2/500 um	2 (SiPIN)	Si	4.4	500	0.00	0.15	Be	Varies	0.0	1.0	155
SiPIN	13 mm2/500 um	2 (SiPIN)	Si	11.1	500	0.00	0.15	Be		0.0	1.0	190
SiPIN	25 mm2/500 um	2 (SiPIN)	Si	21.5	500	0.00	0.15	Be		0.0	1.0	200
CdTe	9 mm2/750 um	3 (CdTe)	Cd	9.0	750	0.15	0.15	Be		0.0	1.0	500
CdTe	25 mm2/1000 um	3 (CdTe)	Cd	25.0	1000	0.15	0.15	Be		0.0	1.0	600

The "window thickness" could be 8 μm (1/3 mil), 12 μm (1/2 mil), 25.4 μm (1 mil), or for CdTe 100 μm (4 mil). This is listed on the data sheet supplied with the unit.

3.1.3 Geometry: The plot on the left defines the key geometric quantities, while the table on the right lists the values for Amptek's standard base plate. Most users have no "optic" so these distances should be set to zero.
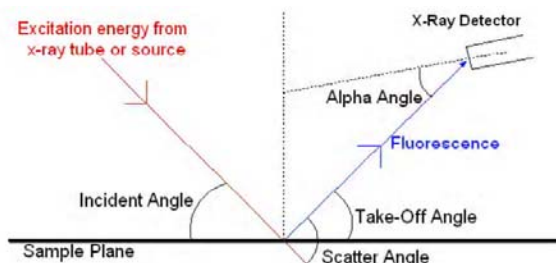
Sample to Detector Distance	1.59	cm
Tube to Sample Distance	3.39	cm
Incidence Angle	67.5	deg
Take-Off Angle	67.5	deg
Scatter Angle	135	deg
Alpha Anlge	0	deg

3.1.4 Tube/Source: This defines the excitation source, whether a tube or an isotope.

- For an isotopic source, e.g. ^{109}Cd, select "Isotopic Source" from the Source Type pull-down menu. Double click on the Source File Name box and call up the appropriate file. With a radioactive source none of the other parameters are relevant.

- For an X-Ray tube, fill in the correct parameters. The table below shows parameters for Amptek's X-ray tubes (and the most common isotopic sources).

| Excitation Source	Source Type	Source Model	Source File Name	Target Element	Target Thickness	Target Incident Angle	Target Take-Off Angle	Window Type	Window Thickness	Optic Type
					(um)	deg	deg		um	
Mini-X Ag	3	Pella		Ag	1.5	90	90	1 - Be	500	1 - None
Mini-X W	3	Pella		W	1.5	90	90	1 - Be	500	1 - None
Eclipse 4	3	Pella		Rh	2.3	90	90	1 - Be	250	1 - None
^{57}Co	4	Spectrum	Co57.txt	Fe	N/A	N/A	N/A	3 - Windowless		1 - None
^{109}Cd	4	Spectrum	Cd109.txt	Ag	N/A	N/A	N/A	3 - Windowless		1 - None

3.1.5 Processing

1. Set the "Peak Width" to match the measured 5.9 keV resolution. Set the "Low Pass Filter Width" to about 1.5 times this resolution. The spectrum supplied with the system shows the resolution measured at the factory, using factory configuration settings. If you have changed the configuration, it is best to measure it again.

2. Set the "Time Constant" to the peaking time used by the PX4 and the "Pulse pair resolution" to 0.6 usec.

3. If you select "Nonlinear Deconvolution," then the software adjusts the centroids and peak widths to get a better match. It often provides a more accurate result, but not always, and it always runs more slowly.

4. Ensure that "Auto Adjust On Load" is checked (for the analysis described here).

5. The other parameters are described in the FP manual.

3.1.6 "Reports" is used to enter data to be printed on the reports generated by XRS-FP. "Reference" is used for advanced analyses.

3.1.7 Spectrum Adjust: In the "Offset" and "Gain", enter the values computed in ADMCA, using its calibration menu. Note that the units are eV in the Spectrum Adjust menu of FP, but the units must be keV in ADMCA.

3.1.8 Quant: For the quick start processing described here select "Fundamental Parameters" and "Standardless." Much more accurate results can be obtained by using standards; please refer to the FP documentation for using standards to calibrate the analysis.
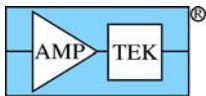
3.2.   Other Parameters

3.2.1   If you are using an X-ray tube then in the "Measurement Conditions" Table of the main screen, enter the voltage (kV), the current (uA), and the filter parameters, e.g. Al for the material and 1000 μm for the thickness.

3.2.2   Near the bottom of the screen click the "Processing" button. Verify that "Escape Peaks" and "Sum Peaks" are checked. Enter the number of times the spectrum should be smoothed.

3.3.   Define the sample

3.3.1   Select the elements.

The analysis will only be applied for elements which are selected and listed in this table. Up to 30 may be chosen, but we recommend limiting them to those which are suspected of being present in the sample.

To remove an element, click on the appropriate row of the "Specimen Component Table" and then click "Delete" on the keyboard. To add an element, click on any row of the "Specimen Component Table" and then click "Insert" on the keyboard. Type in the two letter abbreviation for the element.

By default, the software uses a Gaussian intensity model and no ratio. We recommend using the defaults for the initial analysis. Please refer to the FP manual to change these.

3.3.2   Define the sample geometry.

The "Thickness Information" table permits one to define multiple layers, to obtain results in units of mg/cm2, etc. Please refer to the FP manual for these advanced settings. For the initial analysis, we recommend using a thick, homogeneous sample and obtaining normalized results, thus yielding percent concentration. Set "thickness" to zero, "type" to bulk, and check "normalize."

3.4.   Optional: Save the set-up. Select "File" -> "Save", and select "OK" to replace "MASTER.TFR," the default configuration file. You can give it a different name, e.g. TEST.TFR, and then when you open XRS-FP, open TEST.TFR to recover these settings.

4. Acquire the spectrum to be analyzed

4.1.   Turn off the X-ray tube.

4.2.   Put the sample in front of the detector and source. We suggest beginning with a standard metal alloys. Stainless steel 316 is available in most hardware stores, its composition is well known, and all elements are easily measured by XRF. Brass, with Cu and Zn lines, is also useful.

4.3.   Using ADMCA, acquire a spectrum. The more counts in the spectrum, the better the analysis.

4.4.   Stop acquisition, save the file, then reopen the file, then clear the "live data." FP always operates on a reopened file, not the "live data."

4.5.   See Loading A Spectrum for additional information.

5. Run the analysis

5.1.   Load the spectrum from ADMCA into FP

- Click "Load" on the FP file menu, then click "Spectrum" in the dialog box
- This will load the file which has been read back into ADMCA, not the "Live Data."
- The software will "adjust" the energy scale of the spectrum to obtain a predetermined value, e.g. 20 eV/channel. A shifted spectrum will be visible in ADMCA.
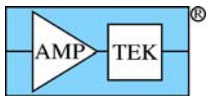
5.2. Process Spectrum

- Click "Process" on the file menu, then "Spectrum." This opens up a box with many options.

- The processing proceeds through the steps listed. If you want to see each step and its result, then click one by one on each step. We recommend doing this the first time to see what the processing does. If you click on "All," it will run through all the steps and then show the result. It uses the settings in the "Measurements and Processing Conditions" table to define the processing steps.

- The result is a processed spectrum appearing back in ADMCA. This spectrum has been smoothed, background has been removed, escape peaks removed, and a Gaussian has been fit to the peaks.

- See Processing for further information.

5.3. Analyze

- Click "Process" on the file menu, then "Analyze."

- The results of the analysis will appear in the "Specimen Component" and "Element" tables.

- The result may be saved and/or printed. To save, go to the "File" menu and click "Save As", then give it a unique name, e.g. "Sample 403.TFR". The .TFR files can be imported into Excel.

- Select "File" -> "Print." The result is a report giving the concentration of each analyzed element in weight % and mole %.
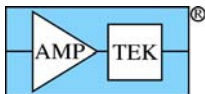

Congratulations! You have now set up the system and carried out an initial analysis. At this point, you can measure different samples or adjust parameters to see what affect they have.

Things to try next

Calibrate the analysis parameters with standards

One of the most significant things you can do to improve accuracy is to perform an "elemental calibration". After the spectrum has been acquired and processed (background removed, escape peaks corrected, and the peaks fit), the result is a table of the intensities of the selected elements. The software then "analyzes" these, using parameters which relate these intensities to the concentrations of the elements in the sample. "Standardless" analysis uses simple models for the spectrometer response to estimate these parameters. Much more accurate results can be obtained if the response is measured for a particular geometry, tube spectrum, and for samples with properties similar to those in the unknown samples which are to be measured. The parameters are then calibrated from the known sample.

The table below shows the advantages of calibration. This shows results measured for two steel alloys, both standard reference materials from NIST, with certified values shown. These were measured in Amptek's system using a standardless analysis, then the parameters were calibrated using a single stainless steel alloy of known composition (a different SS316 reference), and then were measured using the calibrated parameters. The calibrated results are in much better agreement! Twenty measurements were taken, and the STD DEV column shows the standard deviation of the result (the precision). The final column shows an estimate of the measurement uncertainty, produced by XRS-FP. The calibrated results are within measurement uncertainty of all of the NIST results.

		NIST value			Standard less	Calibrated	Std Dev	Uncert
	Cr	18.45	+	0.05	19.00	18.50	0.10	0.25
	Mn	1.63	+	0.01	1.82	1.67	0.07	0.07
Steel 316	Fe	65.19	+	0.07	64.39	65.00	0.18	0.52
	Ni	12.18	+	0.05	12.20	12.27	0.16	0.27
	Cu	0.169	+	0.001	0.137	0.167	0.021	0.026
	Mo	2.38	+	0.01	2.46	2.39	0.02	0.03
	Cr	17.4	+	0.1	17.78	17.59	0.10	0.23
	Mn	1.80	+	0.05	2.09	1.80	0.07	0.07
Steel 321	Fe	69.31	+	0.15	68.34	68.91	0.14	0.53
	Ni	11.2	+	0.1	11.53	11.42	0.16	0.27
	Cu	0.121	+	0.001	0.094	0.119	0.021	0.022
	Mo	0.165	+	0.005	0.177	0.167	0.005	0.009

Calibrating the parameters is described in some detail in Amptek's "Advanced Analysis Suggestions" guide. Briefly, you should (1) under Setup/Quant, select "one standard" or "multiple standards"; (2) obtain a spectrum for a standard, e.g. a known alloy; (3) process it (as above); (4) then, in the Specimen Component Table, enter the known elements and concentrations and select "Fixed" instead of "Calc" under "type"; (5) Select "Calibrate" -> "Spectrum". Repeat 2-5 for however many reference samples you need. Then, and this is critical, get a sample containing all the elements of interest, select "Process", and then "Analyze". If the calibration runs correctly, then in the Element Table (middle of the main screen), clicking the Coefficients button will show a table and the calibration constants will be nonzero.

Optimization

The settings suggested here are really a starting point for tuning and optimizing the system for a particular application. No two XRF applications are the same. Factors such as how many elements must be analyzed, the spacing of the lines, the permitted measurement uncertainty, the required acquisition time, etc. all lead to different optimum conditions. A system to quickly detect the presence of lead on a simple substrate will be very different some a system determining the composition of jewelry! Amptek has an application note suggesting some of the key considerations when optimizing a system and this may provide some guidance. In general, we suggest the user empirically adjust parameters and observe the effects of total measurement uncertainty.
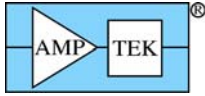
Set-Up Guide

Figure 1 illustrates the key components in a typical setup. Mechanical alignment of the excitation source (either a tube or isotopic source), the detector, and the sample are critical. We strongly recommend a fixture to achieve a reproducible geometry. Radiation shielding (which must be supplied by the user) is critical for radiation safety. The Amptek MP1 mounting plate is an easy way to get a basic fixed geometry.

The most important components are the XR100 detector and preamplifier, the PX4 digital pulse processor, and an excitation source. This drawing illustrates a Mini-X X-ray tube with compact power supply and USB controller, available from Amptek, but many other excitations sources may be used. If using the Mini-X, install a collimator and filter, as describe in the Mini-X manual. Set up must include installing the software on the computer. This includes the ADMCA software, the XRS-FP software, and the Mini-X software. Installation of each package is described in the respective "Quick Start" guide.
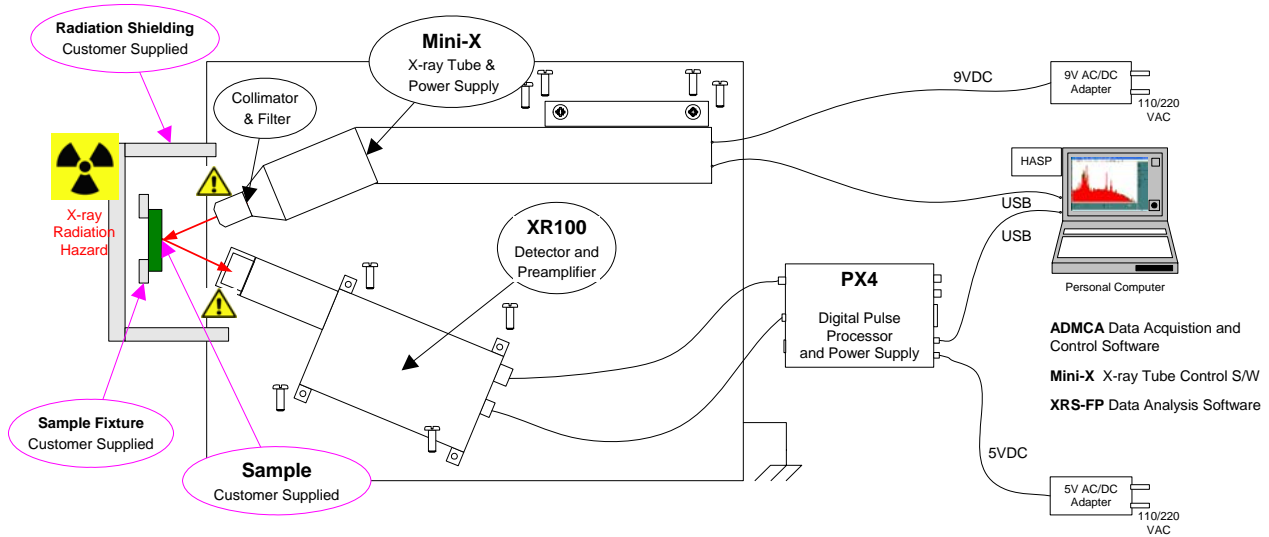
Figure 1. Drawing illustrating a typical experimental setup.
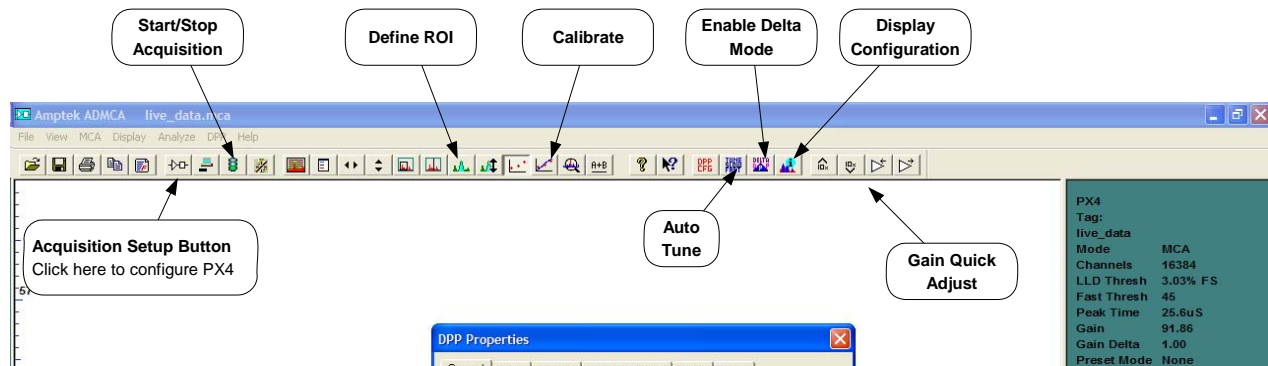
Figure 2. Drawing illustrating the key controls in Amptek's ADMCA software.

The buttons across the top shown here are the most important for the steps discussed here. The "Acquisition Setup" button permits access to all PX4 configuration controls and options. The "Gain Quick Adjust" buttons on the right let one quickly scale the gain. It is convenient to enable delta mode during initial set-up and check-out, and we recommend using "auto-tune" once all parameters are set.

"Start/Stop Acquisition" is clearly important The "Define ROI" and "Calibrate" buttons are used to perform an energy calibration. Once a spectrum is acquired, the data can be saved and the file reopened using the Windows standard Save/Open button. For additional information on the buttons and controls, and on the menu options, please refer to the online "Help" (F1).
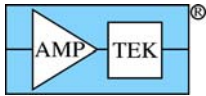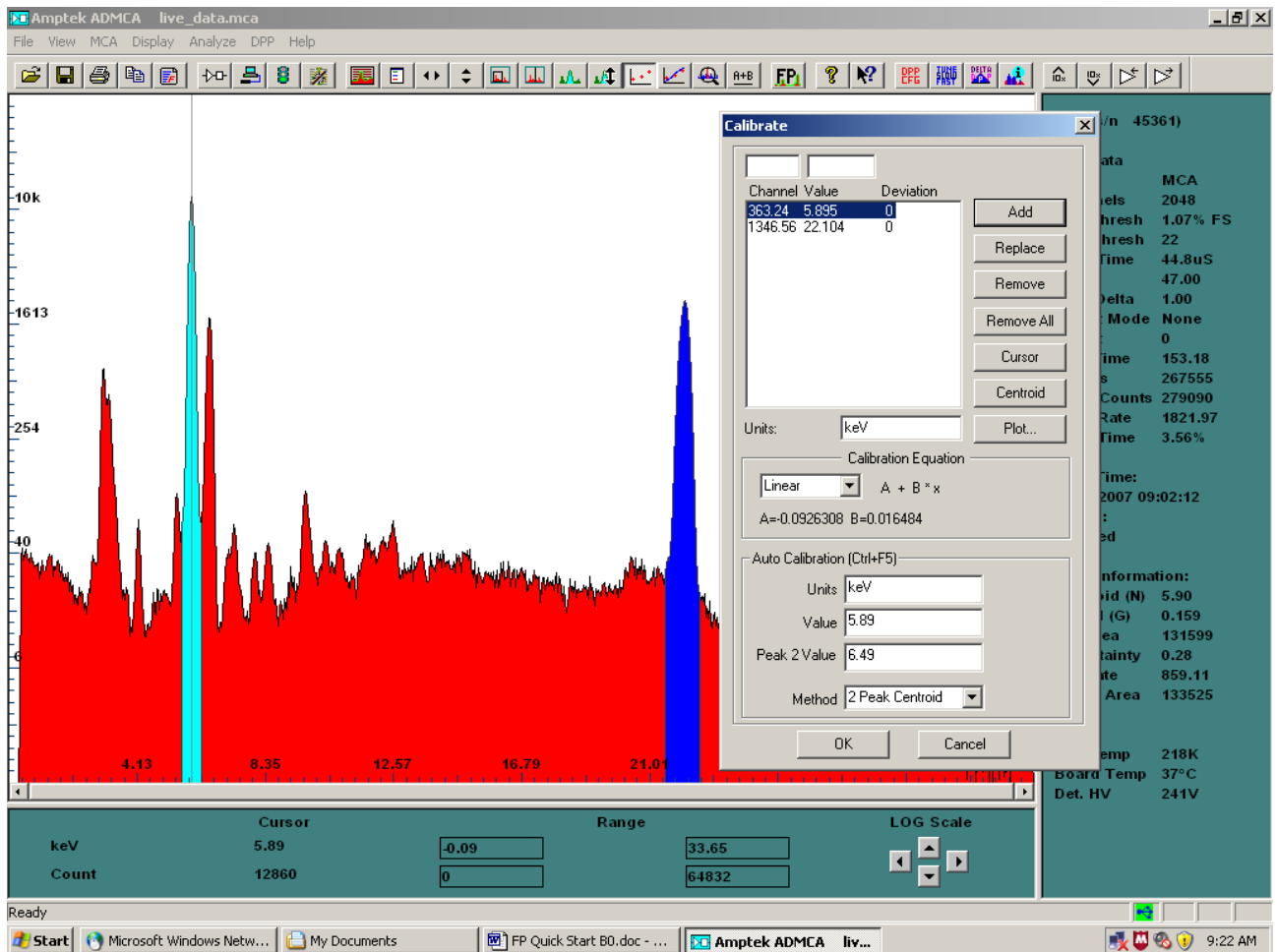
Energy Calibration

Figure 3. Screen capture showing energy calibration in ADMCA.

To carry out a calibration:

1. Acquire the calibration spectrum. In Figure 3, a 35 kVp tube excited a pair of targets, one with 99.9% Mn and one with 99% pure Ag.

2. Mark the Regions Of Interest (ROIs) for the reference peaks. This example used the two K_α peaks, shown in blue. The energies of the Ka peaks are published in many places including the Amptek web site.

3. Press the calibrate button, giving the dialog box in Figure 3.

4. Click in the ROI for the first peak (Mn K_α, light blue in the example), then click "Centroid" in the dialog box. The channel number for the centroid will appear in "Channel" space in the dialog box. Then enter the energy in keV (5.895 in the example) in the "Value" space. Click Add.

5. Click on any additional ROIs and repeat. At least two points are required. If additional points are entered, use the "linear" options and it will perform a linear regression. Enter "keV" in the units box, then "OK".

6. On the main screen, click the "Enable Calibration" button to see the energy scale displayed.

7. In the "Calibrate" box, A is the offset and B is the gain. These are in units of keV and keV/channel, respectively.
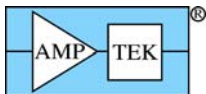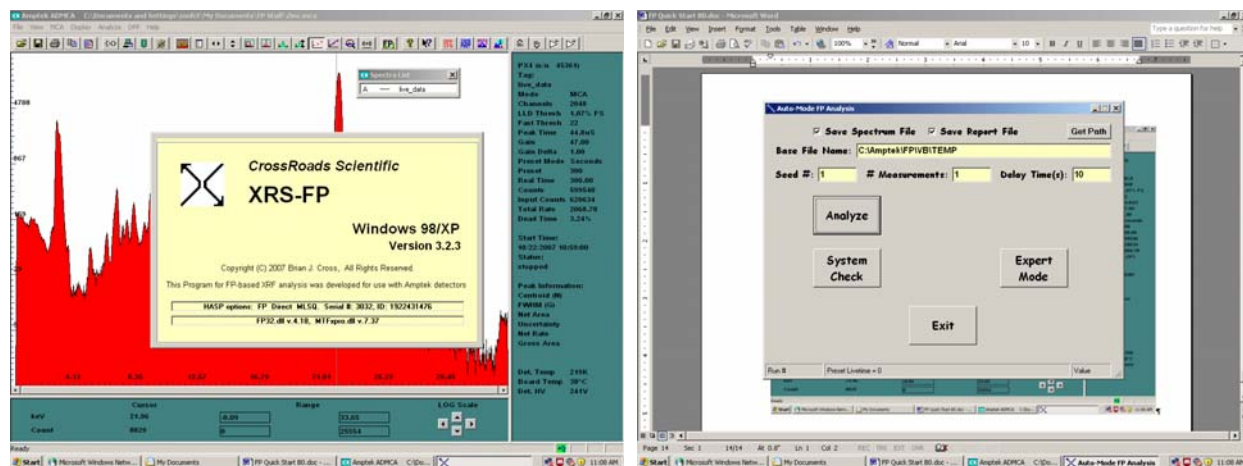
Launching XRS-FP

Figure 4. Screens which appear as FP starts up. Click on the yellow box (left) and "Expert Mode" (right).
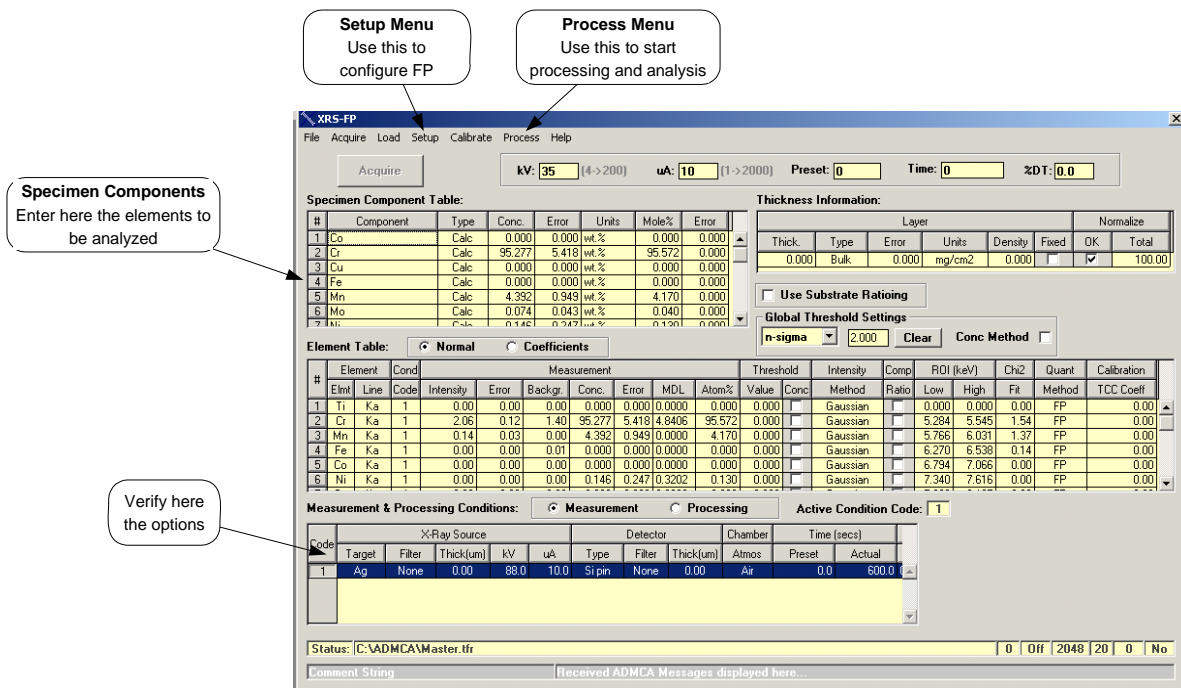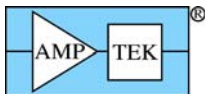

Figure 5. Screen capture of main FP screen, showing the key items.

Loading A Spectrum

The following charts illustrate the processing of an alloy, in this case a mix of Ni, Fe, Cr (about 30% each) with 6% Mo and trace amounts of other elements. The spectrum was acquired for 300 seconds, using the preset value in PX4 Setup. These data were taken using a silicon drift diode (SDD) at 11.2 μsec peaking time.
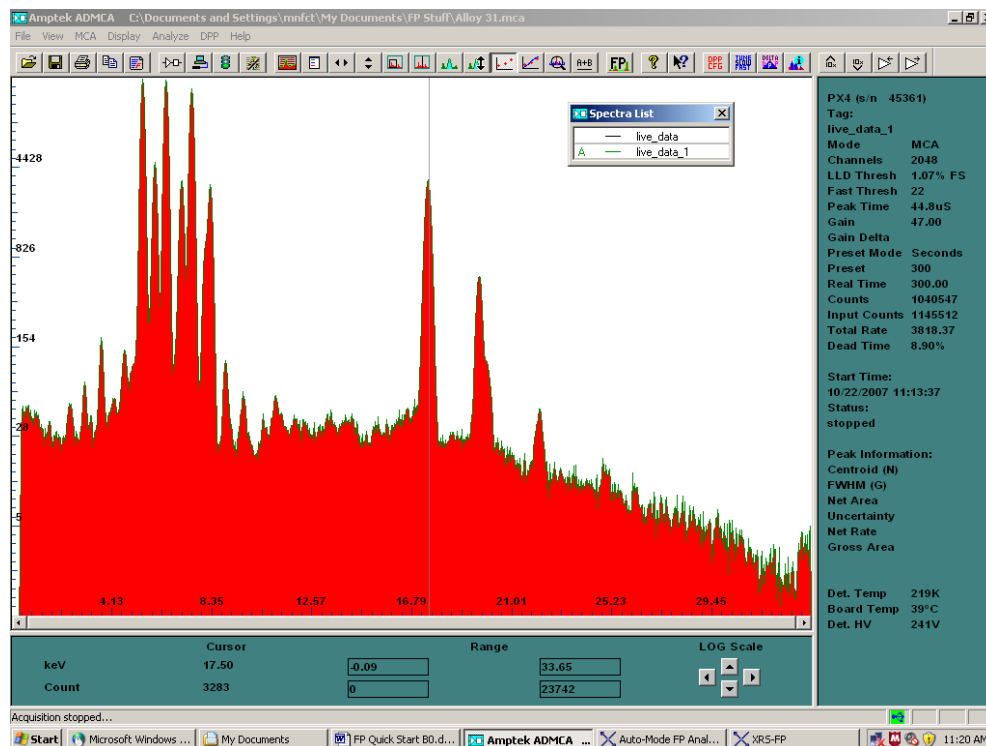

Figure 6. Screen capture from ADMCA after spectrum was acquired, saved, and file reopened. Press "F6" to open the "Spectra List." Once you have reached this point, go back to FP and click "Load" -> "Spectrum."
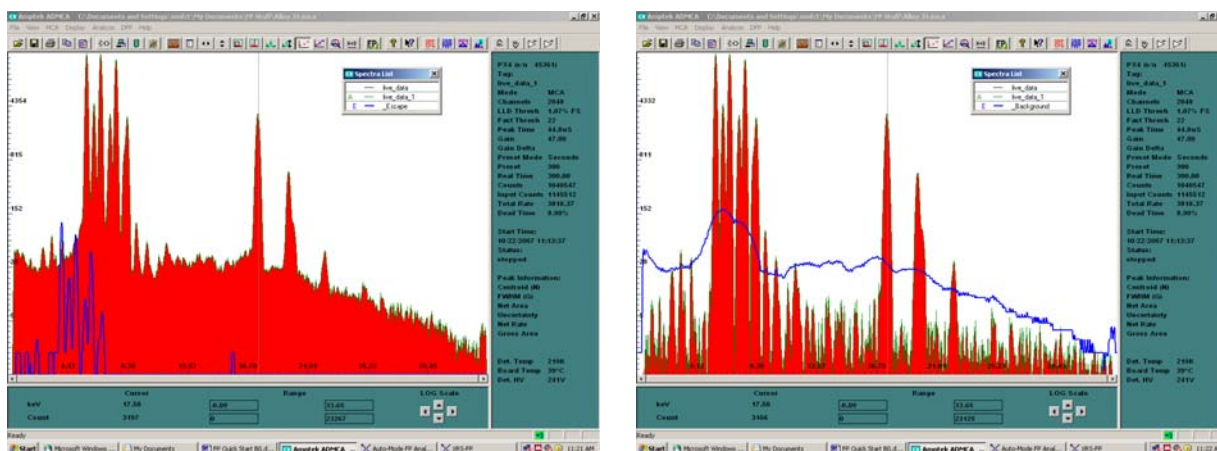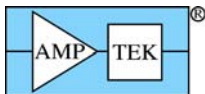
Processing

Figure 7. Spectra showing the results of some intermediate processing steps. Left: After smoothing and escape peak removal. The escape peaks can be seen in blue. Right: After removal of the scattered continuum (in red), with the continuum shown in blue. One can choose to view these intermediate spectra or to let the software pass through to the end result.
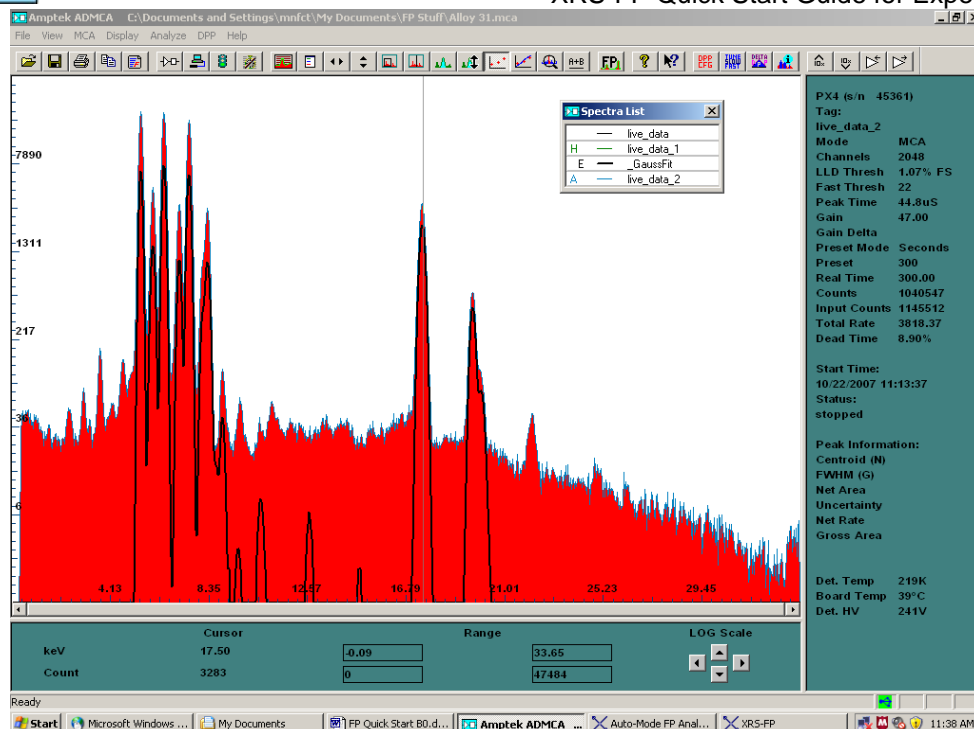
Figure 8. End result of processing, shown as black trace, compared with the raw spectrum in red. The end result is a series of Gaussian functions, fit to the peaks listed in the element table and remaining after the prior processing steps.
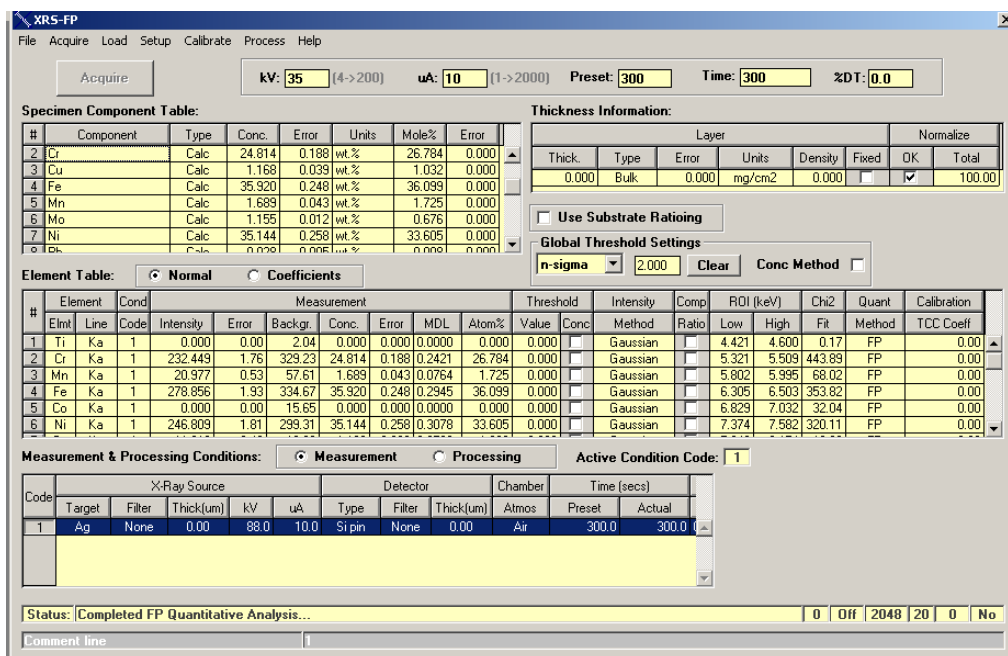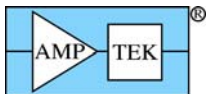

Figure 9. Main FP screen, after quantitative analysis. The area in the Gaussian peaks has been processed to determine the quantity of each element in the sample. This is shown in the "Specimen Component Table", i.e. Cr is measured as 24.8% concentration (by weight) and 26.7% (molar).

XRF Thin-Film Report File
Alloy 31

Sample Table ==
10

Layer	Component	Type	Conc.	Conc.Error	Units	Mole.Conc.	Mole% Err.
1	Co	Calc	0.0	0.0	wt.%	0.0	0
1	Cr	Calc	24.8	0.2	wt.%	26.8	0
1	Cu	Calc	1.2	0.0	wt.%	1.0	0
1	Fe	Calc	35.9	0.2	wt.%	36.1	0
1	Mn	Calc	1.7	0.0	wt.%	1.7	0
1	Mo	Calc	1.2	0.0	wt.%	0.7	0
1	Ni	Calc	35.1	0.3	wt.%	33.6	0
1	Pb	Calc	0.0	0.0	wt.%	0.0	0
1	Ti	Calc	0.0	0.0	wt.%	0.0	0
1	Zn	Calc	0.1	0.0	wt.%	0.1	0

Figure 10. Data file saved from FP, opened in Excel.